WORLD JOURNAL OF PHARMACEUTICAL RESEARCH
SJIF Impact Factor 8.453
Volume 14, Issue 5, 1357-1375. Research Article ISSN 2277–7105

FORMULATION AND CHARACTERIZATION OF HERBAL LIP BALM: A NEWAPPROACH

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Article Received on
16 January 2025,

Revised on 05 Feb. 2025,
Accepted on 26 Feb. 2025

DOI: 10.20959/wjpr20255-35802

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ABSTRACT

Indian herbs and their significance are worldwide accepted. Herbal cosmetics are in increasing demand on the global market because of their fewer side effects, cost-effectiveness, and easily available. Although today trends towards natural living style, including cosmetics. Lip balm formulations are the most frequently used cosmetic items to accentuate the natural way to maintain and promote healthy lips. Currently, cosmetic lip products are based on the use of enormous chemical ingredients with various side effects. Hence in this research project, an attempt has been made to study the herbs that are (Licorice, Chamomile, and Pomegranate) used to formulate herbal lip balm. The Herbal lip balm was formulated using naturally occurring base, oil, extract, colour, and flavoring agents which can be evaluated for their resistance to temperature variations, pleasant flavor, and smoothness during the application, adherence and easy intentional removal, etc. This formulation of herbal lip balm is mainly focused on lip whitening, healthier, and hydration.

KEYWORDS: Herbal cosmetics, Licorice, Chamomile, Pomegranate, Lip whitening.

INTRODUCTION

Indian herbs and their significance worldwide are accepted. Herbal cosmetics are in increasing demand in the global market, due to their lack of adverse effects, cost-effectiveness, and availability. The effectiveness of herbal formulations and their comparative lack of adverse effects, when compared to synthetic medications, have long drawn significant attention.[1][2] For centuries, Indian women have employed henna to dye their hair, palms, and soles, sandalwood and turmeric for skin treatment, and various natural

oils and plants to perfume their bodies.[3] Extensive herbal beauty treatments were once performed in India's royal palaces to enhance sensuality and preserve general hygiene.[4] The herbs possess a variety of pharmacological activities such as anti-bacterial, anti-seborrheic, anti-inflammatory, antiseptic, emollient, and antioxidant activity. Individuals' health, habits, and routine have an impact on their skin and hair's beauty.[5]

Cosmetics play a significant role in today's lifestyle. Cosmetics are substances that are used topically by a person to cleanse, beautify, enhance attractiveness, and change the appearance without altering the body's structure or functions.[6] Moreover, the current trend is going green in almost all industries including cosmetics to adopt a more natural way of life. Formulations including skin protection, sunscreen, anti-acne, anti-aging, and anti-wrinkle products are manufactured for different skin conditions employing a variety of components, either natural or synthetic.[2]

Along with all cosmetic products, Herbal lip balm formulations are most widely used to increasethe beauty of lips and add a glamor touch and shine to the beauty. Lip balms provide a natural means of promoting healthy and moisturized lips.[8] Lip coloring is an age-old method of enhancing lip beauty and adding radiance to the face. To enhance the beauty of lips we have used herbs of Roots, flowers, and fruit i.e., **licorice, chamomile, and pomegranate**. The Glycyrrhiza genus belongs to a family of leguminose comprising more than 30 species. Glycyrrhiza extracts are widelyimplemented in cosmetic products for their good whitening effect. Glycyrrhiza along with its cocosmeceutical activities are categorized as skin anti-aging, photoprotective, hair care, and anti-acne.[9]

In Glycyrrhiza extracts the three main active constituents are licochalcone A, Glabridin, and dehydroglasperin which are flavonoids out of these Glabridin shows the effect of skin whitening.[9] Pomegranate is a plant-rich antioxidant substance preventing skin aging. Pomegranate, containing flavonoid and polyphenolic compounds, is widely used in cosmeceuticals production. Hence ellagic acid, a flavonoid constituent of pomegranate, provides a skin-whitening effect.[10] Chamomile(Matricaria chamomilla) is a widely used herb in traditional use. Chamomile has numerous phytoconstituent with numerous pharmacological actions. Alpha bisabolol is a main activeconstituent that provides a skin-whitening effect.[11] This research project is aimed to formulate anherbal lip balm by using the three-herb licorice, pomegranate, and chamomile that enhanceshealthier and brighter lips. The darkness of lips caused by:[12] Excessive exposure to the sun, Lack of hydration, Cigarette

smoking, Allergic reactions to toothpaste, lipstick, More melanin causes a darker lip tone, Excessive use of coffee. The purpose of this formulation is to provide a healthier, brighter and more hydration to lip for the person having darker lips by using extract of rhizome, fruit and flower i.e., licorice, pomegranate, chamomile respectively.

Difference between lip and regular skin structure.[12]

Lips are more appealing than ordinary skin. The top corneum layer of normal skin typically includes 15 to 16 layers, primarily for protective reasons. In comparison to the skin of the usualface, the top corneum layer of the lip has just three to four layers and is quite thin. There are hardly any melanin cells in the skin of the lips. As a result, the blood vessels on the lips' skin can be seen more clearly, giving the lips their attractive pinkish color. Both sweat glands and hair follicles are absent from the skin of the lips. As a result, it does not contain body oil or perspiration to protect the lip from the environment for these reasons, the lips dry out faster and become chapped more easily.

MATERIALS AND METHOD: Materials

Licorice, Chamomile and Pomegranate were purchased from Yucca enterprises, Neutra Ved Xpotim enterprises and local vendor respectively. All three herbs authenticated from Sonopant Dandekar arts, V. S Apte Commerce and M.H Mehta Science College. All analytical grade solventswere used.

METHODOLOGY

Physicochemical analysis[48][49]

Determination of ash value

Total ash: Weigh and ignite the porcelain dish for 10 min. Take out and cool the crucible Then weigh the empty crucible Weigh 2g of powder, and place the crucible again in the furnace with the powder for 30 min. Take out and cool the crucible; weigh it. Then calculate the total ash value.

Acid insoluble content: Add 25 ml of hydrochloric acid to the crucible containing the whole ash, cover with a watch glass, and gently boil for 5 minutes. Add 5 ml of hot water to the watch-glass to rinse it, then pour this liquid into the crucible. Collect the insoluble matter on an ashless filter- paper. Transfer the filter paper holding the insoluble material to the original crucible, let it dry on a hotplate, and then light it up until the weight becomes constant. After 30 minutes of cooling in a suitable desiccator, immediately weigh the residue. Determine

how much acid-insoluble ash there isin each mg per gm of air-dried material.

Water soluble content: To the crucible containing the total ash, add 25 ml of water and boil for 5 minutes. Collect the insoluble material on an ashless filter paper or in a crucible made of sintered glass. After a thorough hot water wash, ignite for 15 minutes in a crucible at a temperature not exceeding 450°C. Subtract the weight of the entire ash from the weight of this residue in mg from the weight of total ash. Determine the amount of water-soluble ash in mg per g of the material that has been air-dried. Refer table no.1.

Table no. 1: Determination of Ash value.

Parameter	Licorice	Pomegranate	Chamomile
Total ash	6%	3.5%	11%
Limit	9.3%	4%	10-12%
Acid insoluble ash	3%	3.5%	3.5%
Limit	3%	4%	1-5%
Water soluble ash	1.5%	3%	2.5%
Limit	2.5%	3%	1-5%

Determination of extractive value

Water soluble extractive value: Weigh accurately 4 gm of crude drug in 250 ml of conical flask and add 100 ml of distilled water. Keep it aside for 24 hours. Filter it when a sufficient amount of filtrate is collected. Take 25 ml of filtrate into a thin porcelain dish. Evaporate to dryness on a waterbath in an oven at 105 degree for 6 hrs. Cools in a desiccator for 30 min. weigh it immediately. Calculate the percent yield.

Alcohol soluble extractive value: Weigh accurately 4 gm of crude drug in 250 ml of conical flask and add 100 ml of 90% of absolute alcohol. Keep it aside for 24 hours. Filter it when a sufficient amount of filtrate is collected. Take 25 ml of filtrate into a thin porcelain dish. Evaporate to drynesson a water bath in an oven at 105 degree for 6 hrs. Cools in a desiccator for 30 min. weigh it immediately Calculate the percent yield. Refer table no.2.

Table no. 2: Determination of extractive value.

Parameter	Licorice	Pomegranate	Chamomile
Alcohol-soluble	0.8%	8%	11%
water-soluble	2.1%	9%	8%
Limit	1-3%	1-10%	8-12%

Extraction

Extraction of Licorice

Roots were dried at 40 degree Celsius in an oven and then ground to a fine powder and passed through a sieve with mesh 20.

Soxhlet Extraction

Soxhlet apparatus was used to perform Soxhlet extraction. Exhaustive extract with 90% ethanol was performed by using 10g of glycyrrhiza glabra powdered wrapped in filter paper and impregnated with solvent. Extraction was performed with 250ml of solvent. The extract was then filtered through Whatman No.1 filter paper. The resulting yield was calculated.

Extraction of Pomegranate

The peel of pomegranate was dried in the sunlight for 2-3 days and ground into a powder mixture and passed through the mesh 20.

Hydro-alcoholic extraction: Add a crushed powder to a 1:1 mixture of ethanol and water. The beaker was placed in a boiling water bath, covered with aluminum foil, and left for 2 hours. Filter the solution after two hours, collect the filtrate and store it. Calculate the resulting yield.

Extraction of Chamomile

Dried flower of chamomile was ground into powder mixture and passed through sieve mesh 20.

Maceration: In this process, the grind powder of chamomile is placed in stopped container withwhole of the solvent and allowed to stand for at least 3 days i.e., 72 hours with frequent agitationuntil the soluble matter is dissolved. The mixture is then strained (through sieve / nets). Afterpressing the marc, the combined liquids were clarified cleaned by filtration or by decantation.

2.2) Phyto-chemical Screening[48][49]

Sr.no	Constituents	Test/reagent	Licorice	Pomegranate	Chamomile
1	Alkaloids	Dragendorff's	+	+	+
		Mayer's	-	-	-
		Wagner's	+	-	-
		Hager's	-	-	-
2	Carbohydrate	Molisch	+	+	+
		Fehling's	+	-	-
		Barfoed's	+	+	+
3	Steroids	Liberman's test	+	-	+
4	Terpenoids	Salkowski's test	+	+	+
5	Tannins	Ferric chloride	+	+	+
		Lead acetate test	+	+	+
		Potassium dichromate	+	+	+
6	Glycosides	Bontrager test	+	+	+
		Keller-killani	+	+	+
7	Saponin	Foam test	+	+	+
8	Flavonoids	Shinoda test	+	+	+
9	Amino acid	Ninhydrin test	-	+	+

2.2) Thin layer chromatography (TLC)[45][46][47]

For Licorice[46]: Thin layer chromatography was carried out on a precoated silica gel 60 F254 plate. The mobile phase was optimized using different solvent systems. Final mobile phase was **butanol: water: Acetic acid (7:2:1).** The plates were examined under ultraviolet light at wavelength 254 nm and 366 nm. Refer table no.3

Table no. 3: Optimization of mobile phase.

Mobile phase	Ratio
Chloroform: Glacial acetic acid: Methanol: Water	6.4:2.5:0.9:0.1
Ethyl Acetate:Ethanol:Water:Ammonia	6.5:2.5:0.9:0.1
Methanol: water	7: 3

For pomegranate[45]: Thin layer chromatography was carried out on a precoated silica gel 60 F254 plate. The mobile phase was optimized using different solvent systems. Final mobile phase was **Toluene: Ethyl acetate: Formic acid (5.4:3.6:0.9).** The spots are invisible under UV. Therefore, we used DPPH (diphenyl-picrylhydrazyl) as a spraying reagent. After spraying, the spots are visibleat 254 nm and 366 nm in the ultraviolet light.

Table no. 4: Optimization of mobile phase.

Mobile phase	Ratio
Toluene: Ethyl acetate: Formic acid: Methanol	3:3:0.8:0.2
Toluene: Chloroform: Ethyl acetate: Formic acid	2:6:6:2
Methanol: water	10:1

For chamomile[47]: Thin layer chromatography was carried out on a precoated silica gel 60 F254 plate. The mobile phase was optimized using different solvent systems. Final mobile phase was **chloroform: Toluene (7.5:2.5).** The plates were examined under ultraviolet light at wavelength 254nm and 366 nm.

Table no. 5: Optimization of mobile phase.

Mobile phase	Ratio
Chloroform: Toluene	7.5:2.5
Chloroform:Toluene:Ethylacetate	6.5:0.3:0.5
Chloroform: Acetone	9.9:0.1

Rf value of Licorice, pomegranate and chamomile are 0.49, 0.36 and 0.76 respectively indicate the presence of required constituent i.e., glabridin, Ellagic acid and alpha- bisabolol.

Quantification[51]

Total Phenolic content: 0.5 ml of extract of standard is taken and diluted with distilled water up to 10 ml in a test tube. In another test tube take 0.5 ml of ellagic acid extract and dilute it with 1.5 mlof water along with FC reagent and keep it in dark for 30 min. (Before keeping it in dark add 2.5ml of $Na_2Co_{3)}$ After 30 min, both test tube is taken out and from both of it 1 ml of solution is extracted (stock solution) and it is then taken in another test tube and diluted till 10 ml. These are then taken for UV Spectrophotometer analysis. Approximately 2 ml of each test tube content is taken in cuvette and placed in UV Spectrophotometer and readings are noted down (for both test and standard). The readings are used to find out the unknown phenolic content. The amount of Phenolic content present in licorice, pomegranate and chamomile with respect toGallic acid was 6.91%w/v, 9.68%w/v, 3.775%w/v respectively.

2.3) Uv spectral analysis

For licorice[52]: 1g of *Glycyrrhiza glabra* extract was dissolved in 10ml of hydro alcoholic solution. From this solution 1 ml was taken and diluted to 10 ml with hydro alcoholic solution. The prepared solution was scanned in the range of 200-800 nm on a double beam UV-Vis spectrophotometer. Absorbance maxima was found to be 259 nm.

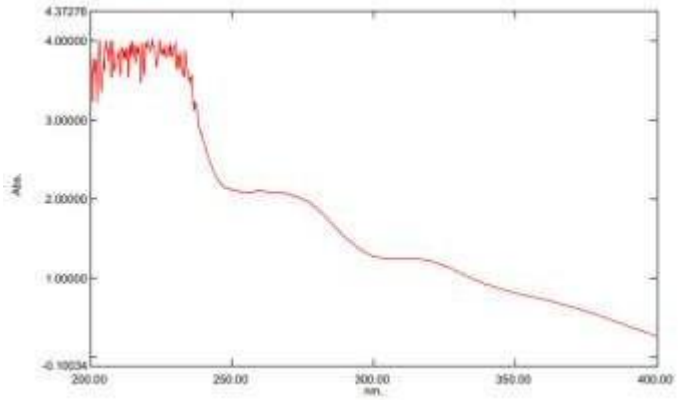

Fig no: 1: Maximum absorption of licorice in hydro alcohol.

UV for Pomegranate[14]: 1g of *Punica granatum* extract was dissolved in 10ml of hydro alcoholic solution. From this solution 1 ml was taken and diluted to 10 ml with hydro alcoholic solution. The prepared solution was scanned in the range of 200-800 nm on a double beam UV-Vis spectrophotometer. Absorbance maxima was found to be 257 nm.

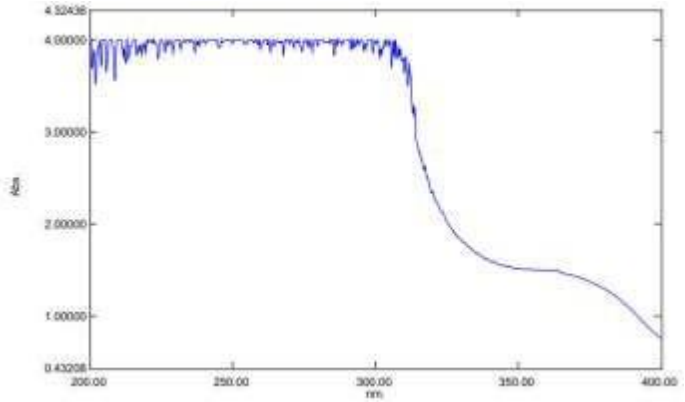

Fig no: 2 Maximum absorption of Pomegranate in hydroalcoholic.

UV for chamomile[53]: 1ml of *Matricaria chamomilla L* extract was dissolved in 10ml of hydro alcoholic solution. From this solution 1 ml was taken and diluted to 10 ml with hydro alcoholic solution. The prepared solution was scanned in the range of 200-800 nm on a double beam UV-Vis spectrophotometer. Absorbance maxima was found to be 264 nm.

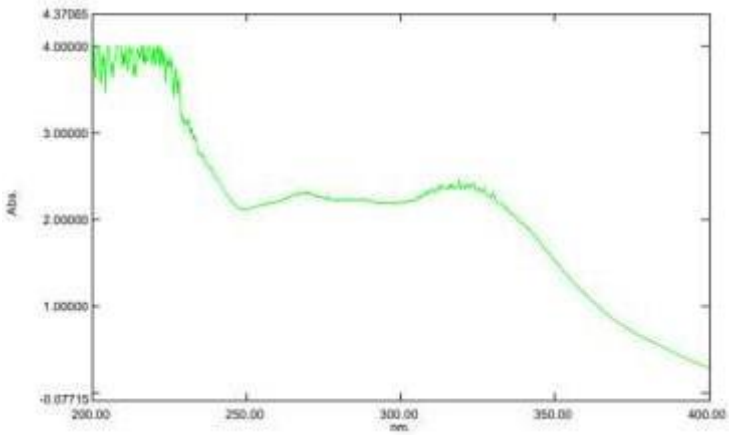

Fig no. 3: Maximum absorption of chamomile in hydroalcoholic.

2.4) Infrared spectroscopy[50]

Infrared spectroscopy, often known as IR spectroscopy or vibrational spectroscopy, is the study of how infrared radiation interacts with matter through absorption, emission, or reflection.

It is employed to examine and characterize chemical elements or functional groups that are present in solid, liquid, or gaseous forms. It can be applied to characterize new materials or to locate and confirm samples, both known and unknown. An equipment known as an infrared spectrometer (or spectrophotometer), which generates an infrared spectrum, is used to carry out the process or technique of infrared spectroscopy. A device used frequently in laboratories that use this method is the Fourier transform infrared (FTIR) spectrometer. The near-infrared, mid-infrared, and far- infrared portions of the infrared spectrum are typically separated into these three categories.

The High energy infrared near approximately 14000-4000 cm-1 The mid-infrared approximately 4000-400 cm-1. The far-infrared is approximately 400-10 cm-1.

Licorice[54]

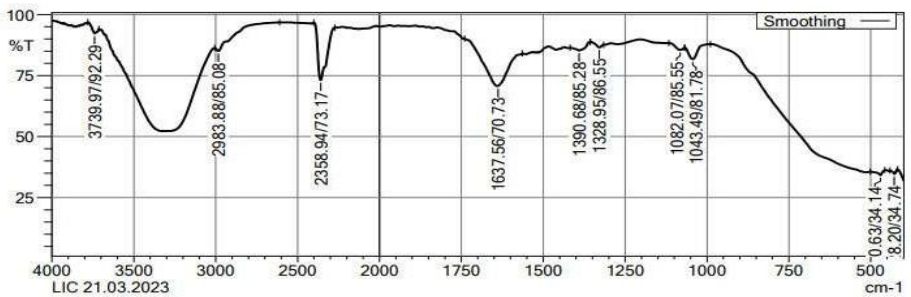

Fig no. 4: IR spectra of Licorice.

Pomegranate[54]

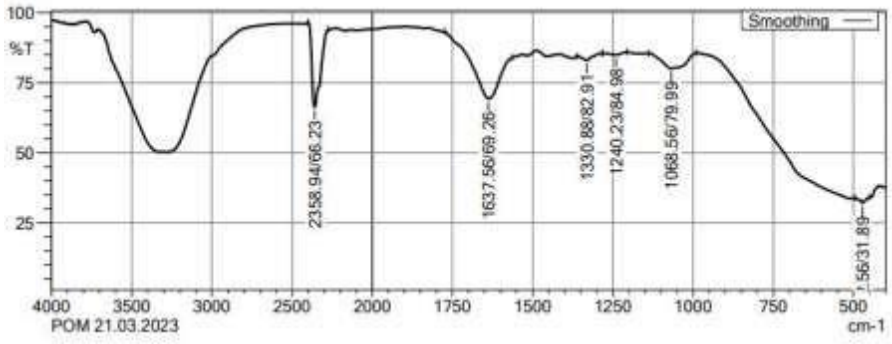

Fig no. 5: IR spectra of Pomegranate.

Chamomile[54]

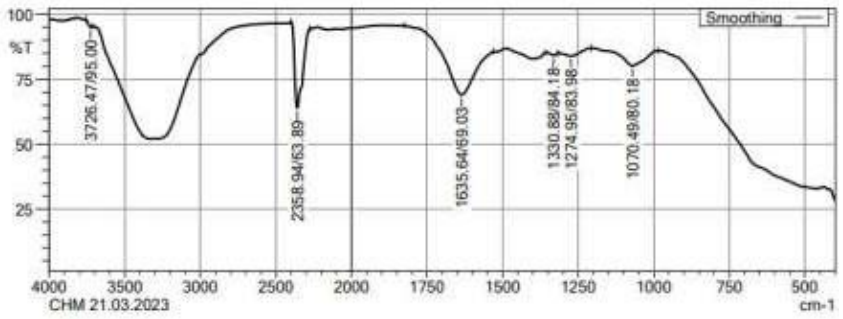

Fig no. 6: IR spectra of Chamomile.

FORMULATION

Procedure

Preparation of Rose oil

Take a clean beaker, add 10 ml of coconut oil into it and 8 to 10 rose petals. Rose petals are properly immersed into the coconut oil. Heat for 30 minutes properly and cool to room temperature. After the cooling, filtrate the solution to get the appropriate amount of rose Oil.

Formulation of lip balm

Weigh all the ingredients accurately. Take the rose oil into the beaker and add herbal extract i.e. Licorice, pomegranate and chamomile and vitamin E oil, boil it for 10-15 Minutes properly until the extract and oil are properly dissolved in the rose Oil. Take another porcelain dish, heat beeswax and shea butter and maintain the Temperature up to 70 degrees Celsius. Mix the oily phase into beeswax and shea butter with continuous stirring and Lemongrassoil at the end. Pour the mixture into a suitable container or lipstick mould. Place the lipstick mould in an ice bath for 10-20 minutes. Keep the lip balm at room temperature for cooling.

Preparation of scrubber

Papaya seed

Take 1-2 gm of papaya seed and dry properly in sunlight. Crush the dried papaya seeds very well. After crushing we used it as a scrubber.

Rose petals

Dried the rose petals in the sunlight for 1-2 days. Crush the dried rose petals properly into mortar and pestle. After crushing we used it as a scrubber.

Formulation table

Sr.No.	Constituent	Formulation batch		
		percentage	percentage	percentage
1.	Licorice	1.5%	1.5%	1.5 %
2.	Pomegranate	2%	2%	2%
3.	Chamomile	1.5%	1.5%	1.5%
4.	Beeswax	50%	50%	50%
5.	Shea butter	50%	50%	50%
6.	Vitamin E	15%	10%	10%
7.	Rose oil	5%	5%	5%
8.	Lemongrass oil	2-3 drops	2-3 drops	2-3 drops
9.	Rose petals (as a scrubber)		0.5 gm	1gm
10.	Papaya seed (as a scrubber)			

EVALUATION OF LIP BALM[29][39]

Evaluation of prepared lip balm was done by checking the organoleptic properties, melting point, pH measurement, test for spreadability and stability Studies.

Test of spreadability

The product is periodically applied at room temperature to the glass slide for the spreadability testin order to visually inspect the uniformity in the formation of the protective

layer. and it is observed that whether the stick fragmented, broke or deformed during the application.

The following standards were developed by the analyst for this test.

G – (Good): uniform, perfect application, no fragmentation, without deformation of Lip balm.

I – (Intermediate): uniform, leaves few fragmentations, appropriate application, few deformation of lip balm.

B- (Not uniform), leaves many fragments, inappropriate application, intense deformation of the lipbalm.

The spreadability of the prepared lip balm was evaluated, and it initially shown high uniformity,flawless application, and lack of deformation at room temperature.

Measurement of pH

Potentiometer (pH) meter

The hydrogen ion activity in aqueous solutions can be measured using a pH meter, a form of potentiometer. The device can display the pH value of a solution's acidity or basicity. The higherthe pH value, the more alkaline is the solution. The lower the pH value, the more acidic the solutionis. It calculates the difference in potential between a reference electrode and a pH electrode. The electrodes in this instrument are rod-like structures. Usually, manufacturers Make these probes using glass. The probe contains a bulb as the sensor at the terminal. The glass electrode specifically contains a glass bulb that is sensitive to the hydrogen ion concentration. When we immerse the probe in the test solution, the hydrogen ions in the solution are exchanged with the positively charged ions The glass bulb generates an electrochemical potential as a result. This potential difference can then be detected by the electrical amplifier and converted into pH units. In order to check for any negative effects, the lip balm's pH was measured. It was made clear to keep the formulation's pH as close to neutral as possible because an acidic or alkaline pH may irritate lips. The pH Measurement was studied by dissolving 1gm of sample into 100 ml of water. The pH measurement was done by using a pH meter. The pH of lip balm was near to neutral pH i.e., 7.04 this would not cause any irritation to lips.

Preliminary Stability Studies

The duration from the formulation's date of manufacturing and packing until its chemical or biological activity is at least at the labelled potency and its physical features have not significantly changed is known as the formulation's stability study. The main aim of stability

study is to provide evidence on how the quantity of drug or drug product varies with the time under the influence of the variety of environmental factors such as temperature, light, humidity. Stability studies were carried out for 15 days at room temperature & refrigeration. The formulated batches were subjected to stability studies for a period of 15 days at room Temperature 25±3 degree and at refrigerator temperature 5±3 degree were found to be stable. Refer table no 6.

Table no. 6: All results are presented in table.

Temperature conditions	Color	Odour	Melting point	Spreadability	PH
25 ± 3 degree	Faint yellowish	Pleasant	68-69.5	Good	7.04
5 ±2 degree	Faint yellowish	Pleasant	68-69.5	Good	7.01

Melting Point

The melting point of lip balm was determined as the melting point sample of lip balm was taken in a glass capillary whose one was sealed by using a flame. In the melting point device, which had a magnetic stirring facing, the capillary holding the formulation was submerged in liquid paraffin. Melting point was determined visually and reported. Melting point of lip balm was found to be in the range of 66 – 68 degree Celsius, which matches with the appropriate melting point of between 65 and 75 degree Celsius. Lip balm's melting point was determined to be between 66 and 68 degrees Celsius, which is inline with the recommended melting point of 65 to 75 degrees.

Organoleptic characteristics

Prepared lip-balm shown faint yellowish coloured with nice aroma. All results are presented in a table.

Table no. 7: organoleptic characteristics.

Parameters	Observation
Colour	Faint yellowish
Odour	Pleasant
Appearance	Excellent, smooth

CONCLUSION

Herbal cosmetics are growing as a result of the enormous demand for beauty enhancing products. The purpose of this study is to provide brighter and healthier lips in the form of

combined herbal extracts i.e., *Glycyrrhiza glabra, punica granatum, Matricaria chamomilla*. Various physicochemical properties of crude drug like Ash value and extractive value were done and the results obtained comply with the standard limit. The phytochemical screening of the herbal crude drugs was performed which serve as the basic to identify the constituent of the extract. The herbal extracts prepared were evaluated qualitatively by performing thin layer chromatography, UV spectroscopy and FTIR which confirms the presence of active constituents responsible for the desired activity. Quantitative estimation of the extracts like total phenolic content were performed. The herbal lip balm was formulated and evaluation parameters like PH, spreadability, melting point, organoleptic characteristic and preliminary stability studies were performed.

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